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DEVELOPMENT OF PROCESSING TECHNIQUES  
FOR ADVANCED THERMAL PROTECTION MATERIALS

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Annual Progress Report for the period  
June 1, 1993 through May 31, 1994.

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## INTRODUCTION

This grant was awarded to the Department of Materials Engineering, San Jose State University, on June 1, 1993. Since then the Principal Investigator, a Research Associate, and several student research assistants have been intensively involved in the research proposed. This report summarizes the progress that has been achieved during the period of June 1, 1993 through May 31, 1994.

A The effort, which was focused on the research and development of advanced materials for use in Thermal Protection Systems (TPS), has involved chemical and physical testing of refractory ceramic tiles, fabrics, threads and fibers. This testing has included determination of the optical properties, thermal shock resistance, high temperature dimensional stability, and tolerance to environmental stresses. Materials have also been tested in the Arc Jet 2 x 9 Turbulent Duct Facility (TDF), the 1 atmosphere Radiant Heat Cycler, and the Mini-Wind Tunnel Facility (MWTF). A significant part of the effort hitherto has gone towards modifying and upgrading the test facilities so that meaningful tests can be carried out. Another important effort during this period has been the creation of a materials database. Computer systems administration and support has also been provided. These are described in greater detail below.

## **FACILITY CAPABILITY EXPANSION**

The Mini-Wind Tunnel Facility, which was designed to produce pressure fluctuations that simulate ascent and reentry conditions of the space shuttle, had not been operated for some time, and needed upgrading. A new electronic control system, which enables not only achieving higher rotor speeds but also measuring the rotor speed accurately, was installed. This effort required establishing system upgrade requirements, identifying suitable electronic components for the upgrade, procurement of the components, installation, and testing of the system as a whole. In this process, new air valves were also installed, so that current facility safety codes are met. This effort has been successfully completed. Tailorable Advanced Blanket Insulation (TABI) materials are currently being tested for their capability to withstand the high aerodynamic fluctuating pressures and aeroacoustic noise levels that they are expected to encounter.

The Radiant Heat Cyclor and the Diffusivity facilities have also been upgraded with new computer control systems and data acquisition systems. The computer control system is necessary to simulate various high heating profiles of future space vehicles. Several heating profiles have been programmed and stored on the computer hard disk drive for easy access during testing. Depending the material being tested, and the test condition to be simulated, different heating profiles can be rapidly recalled, including switching over from one heating profile to another. Test set-up time and turn-around time between tests has now been reduced to a minimum. The electronic data acquisition system has been configured such that the data can be downloaded directly to personal computers, where further data analysis and evaluation can be conducted. More data

points, during testing, can now be acquired and the data reduction speed is significantly faster. The facility improvement here has been tremendous, compared to the difficulty that was being encountered in using chart recorders as the primary means of data acquisition.

The Arc-Jet 2 x 9 Turbulent Duct Facility (TDF) has had its capabilities expanded by the installation of a borescope and a high speed video imaging camera. Installation of the borescope now provides the capability of real-time imaging of test specimens. The video imaging system now provides valuable physical images of the test materials, which can be utilized along with other test data for performance evaluation of materials and designs.

## **HIGH TEMPERATURE RESEARCH**

Current high temperature materials have an operational limit of approximately 1700°C. Advanced high temperature space materials are expected to have to meet at least 3000°C in terms of temperature exposure. Development of these Advanced Refractory Ceramic Materials (ARCM) has included the designing of advanced ceramics that are borides and carbides of the transition metals. These are being screened and characterized in the high temperature 20 MW Arc-Jet Facility. The current effort in this area has been concentrated on designing and building appropriate test fixtures for supporting the specimens and in developing appropriate test specimens and test protocols. Fixtures were designed using a CAD/CAM system, and fabricated out of graphite, in order to meet the extreme thermal shock requirements.

Other techniques that are being used to characterize the ARCMs include thermal dilatometric analysis to determine coefficients of thermal expansion, x-ray diffraction analysis to determine the development or absence thereof of new chemical formulations during processing or testing, density measurements to determine the percentage of open and closed porosity, and sonic moduli testing for flexural data.

Development of appropriate sample preparation techniques for microstructural analysis has been another area of effort. The challenge has been proper preparation of heterogeneous materials which have varying degrees of hardness and ductility within it. The procedure developed involves pre-infiltration of the specimen to protect the oxide layer from handling damage, sectioning the material, encapsulating it in epoxy, and then grinding and polishing it with appropriate compounds to yield the desired grain structures. Image analysis is being conducted with optical microscopy, utilizing both white and fluorescent lighting.

Investigation of solid state reactions between dissimilar ARCM compositions, in a very high temperature environment, is being planned for the near future.

## **ESTABLISHMENT OF MATERIAL DATABASE**

The Thermal Protection Systems Expert and Material Property Database, or TPSX, is a personal computer-based program which serves as a database for advanced thermal protection material properties. The TPSX database program provides an easy user interface for retrieving material property information in a variety of forms - both graphical and text. The database itself consists of a set of text files which can be easily

modified and expanded to include new materials. The primary purpose and advantage of TPSX is to maintain a high quality source of often used thermal protection material properties in a convenient, easily accessible form, for distribution to government and aerospace industry communities.

The TPSX development project evolved out of the necessity for a "centralized storehouse" and retrieval system for materials information and thermodynamic property data. Before this project was initiated, NASA engineers and contractor employees had to spend many valuable hours for specific TPS materials and property information. Creation of the TPSX has greatly reduced the time and effort required to obtain TPS data. TPSX was to be a repository and search tool to assist materials and other engineers in obtaining important reference and property data. From this initial plan, at the beginning of this project, TPSX has matured into a full-featured material database with extensive search capabilities and an analysis input tool able to produce output formatted for computer codes such as CMA, ASC and COSMOS/M.

Work began on this phase of TPSX in July 1993. The main objectives were:

1. To locate and retrieve the extensive NASA-Ames TPS material property information.
2. Develop an interface that will allow a user to search the database for needed information, and choose the format, graphical, text or other, to display it.

In order to accomplish the first objective, a Material Inclusion Survey was distributed. Each of the engineers at the Thermal Protection Materials Branch was asked to fill out as much of the survey as was possible. This preliminary information

sheet included questions such as: material name, distribution restrictions for proprietary materials, type of material, data storage medium and platform, as well as the specific properties which were to be included in the database. Since the survey was an initial attempt at obtaining the needed information, this process was followed up by personal contacts with the NASA engineers. As a final effort to ensure accuracy and completeness for the RTM portion of the database, meetings were scheduled between the Branch Chief and the individual engineers. During these times each scientist was able to get hands-on experience with a beta test copy of TPSX. Sources and examples of some of the database information are listed in Table 1.

Realization of the second objective began when the database files were nearly complete. Application construction involved two parts. First, building of the graphical user-interface and developing the accompanying computer code for executing the search, and second, incorporation of the actual RTM data files into the program. The "Microsoft Visual Basic 3.0 Programming System for Windows - Professional Edition" was used to develop the application. The search routine offers the user two different means of searching the database. The first method is by element components and the second is by specific component properties. For the first method, up to four different elements from the Periodic Table of the Elements may be selected for inclusion in the search. The routine may be executed such that:

1. All compounds including any or all of the selected elements are reported;
2. Compounds containing exclusively the elements selected will be reported.

The second method enables the user to select at most two properties from eight

different materials properties and to specify a numerical range for the value. At completion of the search, a text window displays the compounds, if any, that fit the search criteria. A selection may be made from the text window and the corresponding properties of the material are displayed in the output window. At this time, a user may choose to produce a hard copy printout of the search results. An "output" button on the interface produces a formatted listing of the properties that is fully editable and printable to the Windows default printer.

Version 1.00 of TPSX is receiving the latest additions to the databases and awaiting final approval. Upon distribution of Version 1.00, program debugging and enhancements will continue. A few of the current features of TPSX include a units convertor, engineering analysis code input stack capability, a periodic table of the elements, a physical constants listing, a graph window and a picture window. Some of the future plans for TPSX are: a 1-D thermal analysis module to estimate material temperatures under a given heating condition, a module to predict the heating conditions for atmospheric entry trajectories used in the design of thermal protection systems, and porting the program to other computer platforms such as the MacIntosh.



**Table 1**

RTM engineers	Los Alamos National Laboratory*	PathFinder Program**
LI-900 (rigid tile) FRCI (blanket) AETB (blanket) TUF1 (surface) Silica Aerogel Diboride Composites Carbon Composites	Extreme heat resistant refractory composites	Ablation materials for high energy aerobraking vehicles (Nylon-Phenolics, Silica-Phenolic, Polybenzimidazoles)

\*Vier, D.T., *Thermal and Other Properties of Refractories Technical Report Program No. R056, Los Alamos Sci. Lab., Los Alamos, NM, 1975.*

\*\*Pathfinder Thermophysical Property Data - *Thermal Protection Materials For High Energy Aerobraking Vehicles - Vols. 1-2, Job Ord. #s 80-230, 63-280, Lockheed Eng. and Sci. Co., TX, 1990.*